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Best Wishes for a Merry Christmas and a Bright and Happy New Year

Some Properties of Metal Soap — Mineral Oil Systems

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A great deal has been published on the methods of manufacture and composition of commercial lubricating greases and a good portion of this material has been collected by Klemgard (6). Systematic work on the behavior of soap—mineral oil systems, however, has been very slow in appearing. The first notable contribution in this connection was made by Lawrence (7). His work included a study of gelation phenomena when soaps of the alkali and alkaline earth metals were dispersed in pure hydrocarbon oils. Lawrence demonstrated the presence of a plastic temperature range in most soaps and reported the transition temperatures for a number of these. He also pointed out the importance of small amounts of polar substances such as water, glycerol and fatty acid on the properties of these dispersions. The fact that lubricating greases are not emulsions, was also emphasized.

Any work of a fundamental nature in connection with such a complex mixture as lubricating grease necessitates an examination of the individual components. It is well known in the industry that minor changes in procedure or composition may have a marked effect on the product produced. It is therefore desirable to find out the effect of each variable, if possible. In 1938 a rather extensive investigation of the properties of lubricating greases was initiated at the National Research Council under the sponsorship of the Imperial Oil Ltd. Most of the data presented here were obtained during that investigation and have been published elsewhere in more complete form (1, 2, 3, 4, 5). Since the two most common types of grease used today are those composed of calcium or sodium soaps dispersed in oil, the present paper will be confined to the properties of those systems.

Sodium Soaps

Physical Properties

If sodium stearate is dispersed in a mineral oil at room temperature it will be found that virtually no solution occurs and the soap will settle out in the bottom of the container. If the mix is heated with stirring, the viscosity of the dispersion will at first decrease and then suddenly increase in the neighborhood of 125 °C. The viscosity will then decrease gradually as the mixture is heated further, and finally a clear mobile solution will be obtained, from which crystals of soap may separate, or if the concentration of soap is sufficiently high, a gel will form on cooling. The properties of the crystals and of the gel will be found to be quite different if a highly paraffinic or a highly naphthenic oil is used as the dispersion medium. The viscosity of the oil will have an effect also. Since it appears that a drastic change takes place in the soap at the swelling temperature, a more detailed examination of the soap alone and in combination with oil in this region becomes desirable.

To give more physical meaning to Krafft's rule, which states that in aqueous soap solutions a change in the degree of dispersion takes place at the melting point of the fatty acid, Thiessen and Ehrlich (9) postulated unidimensional melting, i.e. at the melting point of the fatty acid sufficient thermal energy has been supplied to bring about a loosening of the soap crystal lattice in one dimension. This conclusion was reached after measurements of specific volume, specific heat and optical double refraction had been made. Dielectric constant and X-ray measurements also indicated changes at this temperature. Continuation of dilatometric measurements with sodium stearate beyond the temperature range covered by Thiessen and Ehrlich have produced interesting results. Following the initial break at 68 °C. the density falls off sharply until a temperature of 125 °-135 °C. is reached. Here the slope of the curve changes and becomes linear up to 190 °C. where a third break occurs. The essential features of this curve were later substantiated by Vold, Macomber and Vold (5).

Following the terminology of Thiessen, these three breaks in the density-temperature curve have been termed the uni-, bi- and tri-dimensional melting points. Above the bi-dimensional melting point the soap is plastic. This may be noted in a test tube

by its tendency to smear if rubbed with a glass rod, and is to be expected, since the hydrocarbon tails of the soap molecules are free to move in two dimensions and a medium of slip is provided by the "melted" portions of the lattice. Thiessen observed that double refraction in sodium stearate became zero at the plastic temperature threshold. Complete melting is obtained at 190 °C. Further evidence for a structural change in the soap was found by carrying out a differential cooling curve. A sharp break was found again at about 120 °C.

Dilatometric measurements with sodium oleate indicated a sharp break at about 100 °C.

Limited quantities of addition agents such as free fatty acid, glycerol or naphthenic base oil, had little effect on the bidimensional melting point of the soap but seemed to promote solution gradually at higher temperatures, wiping out the complete melting point density change. Free alkali and highly paraffinic oils appeared to act more as inert diluents.

Measurements of the surface tension of 1% dispersions of soap in oils showed sharp breaks in the vicinity of the 100 °C. for both sodium stearate and oleate in a naphthenic oil. With a paraffinic oil, a limited amount surface activity was found at higher temperatures.

The surface relations of various oils and soaps may be demonstrated by other simple experiments. It is well known that a powdered solid shows different equilibrium settling volumes in different liquids. This has been attributed by Ostwald and Haller (8) to the formation of "lyospheres" of different sizes about each particle. These lyospheres were considered to be diffuse adsorbed liquid layers and their size controlled the sedimentation volume.

Equilibrium sedimentation volumes of equal amounts of sodium oleate in paraffinic and naphthenic oils are given in Table I. The volumes were obtained by allowing a suspension of 2 grams of-240 mesh sodium oleate in 50 cc. of oil to come to equilibrium.

TABLE I

Temp. °C.	*Type of Oil*	*Viscosity (S.U.S.)*	*Sedimentation Volume cc.*
25	Naphthenic	900	25.0
	Paraffinic	900	9.0
	Naphthenic	100	9.5
	Paraffinic	100	7.9
80	Naphthenic	900	39.5
	Paraffinic	900	12.9
	Naphthenic	100	18.3
	Paraffinic	100	14.0
100	Naphthenic	900	50.0
	Paraffinic	900	17.0

Based on the data obtained from the density-temperature curves, surface tension measurements and sedimentation volumes, it could be predicted that more stable soda soap greases could be prepared with naphthene than with paraffin base oils. The former produce systems which are much more lyophilic and the tendency for separation to occur would be much less than with paraffin oils of the same viscosity. Viscosity measurements on slurries of this type showed a yield value with the dispersion in the naphthenic oil but no yield value with the paraffinic oil. This may be due to incipient swelling on the surface of the soap particles dispersed in the naphthene base oil. This would cause bridging of the soap particles and definite pressure would be required to cause flow. It would also account for the higher sedimentation volume.

FIBRE FORMATION

Since soda soap greases contain crystals which almost invariably have a high length to breadth ratio, the factors governing their formation are of considerable interest. Fibre length is frequently determined in the industry by the length to which a sample of grease may be pulled out with the fingers. Since the length of macro fibres which can be produced is roughly proportional to the length of the micro fibres present in the grease, this procedure gives a fair indication of the fibre length. Microscopic examination of the soap fibres in the grease may be accomplished either by viewing the sample between crossed nicols or with a cardioid condenser. If the fibres are long, however, a much clearer picture is obtained by dispersing a small portion of the grease in ethyl ether and pouring it over a microscope slide. After washing away the excess oil with more ether, the soap fibres may be viewed with transmitted light at 200 diameters magnification.

It has been found that fairly long fibres may be obtained by dissolving the soap in oil at about 180 °C. and allowing the mixture to cool at about 10 °C. per hour. Polar addition agents such as glycerol and fatty acid or better still the use of a naphthene base oil as a crystallizing medium will increase the length to breadth ratio of the crystals. The use of soaps with unsaturated fatty acid radicals increases this ratio much more. However, crystals of soap formed during grease manufacture are generally produced under conditions of shear, and experiments were devised in which shear was introduced.

Exposing soap-oil dispersions to conditions of shear at ordinary temperatures causes a breakdown in the crystals with a consequent shortening in the micro fibre length. Since the crystals of sodium stearate appear to be more brittle than those containing sodium oleate, the breakdown is slower when unsaturated soap stock is used. As the temperature is raised to about 125 ° C., however, the effect is reversed and not only are the fibres not broken down, but new ones are formed. If, for example, a soda base grease is greatly broken down by working, it can be brought back to its original fibrous condition by heating to 125 °C. and passing through a colloid mill.

A series of experiments in which 10% of soap was dispersed in oil, heated to about 130 °C. and passed through a colloid mill revealed the following information regarding fibre length.

(1) The fibre length increased as the ratio of sodium oleate to sodium stearate was increased.

(2) The fibre length and stability of the product increased as the naphthenic content of the oil stock was increased.

(3) The fibre length increased as the viscosity of the oil was decreased.

During the manufacture of grease most of the oil is added while the soap is in the temperature range for plasticity i.e. above 125 °C. In this range the soap lattice opens up sufficiently to allow oil to penetrate and the system becomes quite lyophilic. Any shear applied at this time will draw out fibres and a structure which enmeshes large quantities of oil will be formed. Since the degree of plasticity of the soap depends on the temperature, the temperature at which the oil is added during grease manufacture will partially depend on the rate of shear available in the grease kettle. Shearing below the plastic temperature range will merely break down the fibrous structure. The formation of macro fibres in grease appears to be due to orientation of the smaller fibres. This may be observed microscopically by incorporating heavier fibres such as sulphite cellulose into a heavy oil and pulling out threads with a micromanipulator. It will be found that this treatment causes the anisodimensional particles to be predominately orientated in one direction with a good deal of overlapping to give a fair amount of rigidity.

LOW TEMPERATURE FORMATION

Since greases consist of soaps properly dispersed in oils, it is conceivable that the dispersion may be accomplished in two ways, namely:—the reduction in size of larger particles, the method usually used in industry, or the formation in the oil of the correct particle size by an agglomeration process. This latter method has been used industrially to a limited extent in the manufacture of cold set greases. It has been found, however, that this process can be extended to sodium or calcium base greases with the usual fatty acids. It is only neces-

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sary to add the theoretical amount of a dispersion of alkali in oil to a portion of oil containing the desired concentration of fat or fatty acid, agitate until the mix is uniform and warm to about 60°C. The reaction is almost instantaneous and the heat of reaction will raise the temperature somewhat higher. The product formed is smooth in texture but if a soda base grease, it may be made fibrous by heating to 125°C and submitting it to a high shearing stress.

The chief advantage of this procedure is the low temperature at which the reaction goes, with the consequent saving of heat and time. Since the dispersion of soap is uniform and fine, the yield of grease obtained is considerably higher than in products with equal concentrations of soaps prepared in the conventional way. Sodium or calcium hydroxide may be dispersed in oil in concentrations of 30 to 40%, with the aid of a suitable mill, to form the stock suspension. The best results are obtained when the alkali is as finely divided as possible.

Although it is stated by Klemgard that fatty acids with a chain length of less than 10 carbon atoms are of no use in grease manufacture, with this "in situ" method of saponification, greases of normal appearance and texture can be made with acids as low as butyric in molecular weight. It would seem probable, since greases cannot be formed in the conventional way using these low molecular weight fatty acids, that the soaps formed from them do not show a bi-dimensional melting point and would not form fibres.

CALCIUM SOAPS

Perhaps the most interesting aspect of calcium base greases lies in the fact that dispersions of calcium soaps are not stable in most mineral oils and usually require a third component to produce stability. Since water is the stabilizing agent most generally used, the definition that a lubricating grease is an emulsion of water in oil stabilized by calcium soaps, has become prevalent. Before examining the properties of calcium soap dispersions in mineral oils, produced at elevated temperatures, it is of interest to investigate some of their surface properties at room temperature, and the effect of water on these properties.

CONTACT ANGLES

It is well known that the angle of contact which water makes with the surface of stearate acid which has been prepared by cooling the molten acid against glass is much smaller than the angle formed with the surface which cooled exposed to air. This is usually attributed to the orientation of the more easily wetable carboxyl group to the polar glass surface. Since calcium stearate, like stearic acid, is highly dipolar, the magnitude of the contact angle of oil against its polar and non-polar surfaces becomes of importance. Table II shows the values obtained for the contact angle of several liquids on calcium stearate.

The surface referred to as moistened was flooded with water and allowed to drain, leaving it apparently dry. The difference in contact angle, however, shows that an important change in properties has been brought about. As expected, the reduction in contact angle with the addition of water is less for non-polar than for polar surfaces. It is interesting to note, however, that the contact angle on both polar and non-polar surfaces is brought to the same value by moistening. No difference could be found between paraffin or naphthene base oils. The wetting process is at least partially reversible, since, when the moistened polar surface was allowed to stand for 18 hours at room temperature before measuring the contact angle, a value of 45° was obtained. It would appear that the small amount of water adsorbed by the polar portions of the surface provides a medium over which the oil spreads much more readily. It is evident from this that water plays an important part in the surface relations of calcium stearate-oil systems.

SEDIMENTATION VOLUME

The effect of addition agents on the equilibrium sedimentation volume of dispersions of calcium stearate in oils is very marked. The addition 0.1 cc. of water to 100 cc. of oil containing 4 gms. of vacuum dried calcium stearate caused the sedimentation volume to increase from 12 to 20cc, while the rate of sedimentation was about doubled. The supernatant liquid was also much clearer. Other addition agents which increased the sedimentation volume were glycerol, stearic acid and alcohol, while calcium hydroxide, acetone, ether, chloroform and

TABLE II

Nature of Surface	*Liquid*	*Contact Angle, degrees*	
		Dry Surface	*Moistened Surface*
Polar	500 visc. paraffinic oil	57	35
Polar	500 visc. naphthenic oil	58	33
Polar	Water	83	....
Non-Polar	500 visc. paraffinic oil	43	38
Non-Polar	500 visc. naphthenic oil	45	34
Non-Polar	Water	113	....

carbon tetrachloride were found to have no effect at all.

Since 0.1 cc. of water is capable of increasing the sedimentation volume by 8 cc. it would seem that Ostwald and Haller's theory of lyospheres is, in this case at least, untenable. A more probable explanation, based on microscopic observation lies in the fact that the sedimentation volume is a measure of the degree of flocculation of the solid. If the attraction of the solid particles for each other is greater than for the liquid medium, the degree of flocculation will be large, the sedimentation time short, and the sedimentation volume large. In such cases a scaffolding structure will be formed and a degree of rigidity produced in the settled volume. If on the other hand the solid particles have no mutual attraction, they can flow freely past each other to occupy a minimum volume. The settling time in such a case will be long and the sediment will have no rigidity. Water and certain other polar compounds seem to be adsorbed by the polar part of the calcium stearate crystals and bring about agglomeration through mutual attraction. The type of oil used as a settling medium appears to have little effect.

Flow-Pressure Relations

Since a structure is formed by the flocculated particles, a yield value should be found if flow-pressure curves are measured. Viscometric measurements on slurries of calcium stearate in mineral oil showed that this was actually the case. The addition of 5% of water, based on the soap, raised the yield value of a 40% suspension of calcium stearate in mineral oil by 15 grams without altering the slope of the flow-pressure curve. The properties of this material after the addition of water were very similar to those of a grease, except that the yield was low for the soap content. The use of calcium oleate, however, instead of the stearate produced quite good greases at room temperature. Increase in the yield value with increased concentrations of water reached a limit at about 7 to 8% based on the soap.

When the temperature of a dispersion of calcium stearate in oil is raised, the viscosity of the mix decreases until a temperature of 100 to 110°C. is reached when the soap suddenly swells and a maximum is obtained, after which the viscosity again decreases as a clear solution is formed. On cooling, the soap crystallizes and the size of the crystals can be partially controlled by the rate of cooling. With paraffinic oils separation of oil and soap takes place, while with naphthenic oils a stable grease may be formed with a low yield value. The addition of water during the cooling, changes the picture, however, considerable flocculation of the soap takes place and if the sample is examined at 200 diameters between crossed nicols much greater contrast between crystals and background is obtained than if no water is present. The system now shows stream double refraction strongly, while this property was virtually absent before the addition of water. This would seem to indicate that the crystals are anisodimensional and are bridged to each other by the adsorbed water. When a stress is applied, the majority of these crystals align themselves in one direction to produce the stream double refraction. Free fatty acid also produces a limited amount of stream double refraction.

It has been found that the amount of water required to produce stability in these systems, varies with the free fatty acid content. For example, when six commercial samples of anhydrous calcium base grease were titrated with water at 95°C. until they were just stabilized, the sum of the percentages of water and free fatty acid varied from 1.2 to 1.5. This would appear to indicate that some minimum amount of water and fatty acid is required for stability. The addition of water also appears to promote crystallization in the swollen soap-oil mixes. This may be observed from the flow-pressure curves, since the swollen soap behaves like a viscous liquid and shows no yield value. The addition of water isothermally changes this by reducing the viscosity and producing a high yield value.

Dilatometric measurements on slurries of calcium soap show a large decrease in density at the swelling temperature, similar to that obtained with sodium soap dispersion and it appears likely that it too goes through a bi-dimensional melting.

The production of calcium soap greases is essentially one of controlled crystallization in which the microscopic crystals are bridged together by the polar stabilizing agent, water. In contrast to soda base greases, the type of oil used has little effect on the product obtained. This is probably due to the fact that water is the controlling factor in stabilizing the system, rather than any relation between the soap and oil.

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Aircraft Engine Oils

(Continued from Last Issue)

Post-war private flying will open new opportunities in the marketing of petroleum products, services, and accessories.

Sales and profits may literally land on the station able to provide suitable landing facilities. Operators may have already gazed upwards, wondering if some day planes flying overhead might land for refilling of the fuel tank and a lubrication job.

Plans for post-war flying indicate this situation may come to pass for stations along main highways, or at resorts, where a landing strip can be laid.

Operators of such combination air-land "super" stations naturally are concerned with the types of products required for the aerial customers. One of these products is oil for the engine crankcase, especially as to oil grades required and the similarity to oil required by automobile engines.

Over 30,000,000 car owners are, to a large extent, familiar with SAE grades of Motor Oils. Many of these owners understand the technical details and specifications while others have only a slight idea what it is all about. But at least most car owners recognize that the lower the number the lighter the oil.

SAE grades for automobile Motor Oils have been in use since about 1926. For the past 15 or 20 years advertising, literature, sales talks, and other things have been built around using the proper grade for the temperature condition. Therefore, it is unlikely that many automobile owners do not know of SAE grades of oil.

The SAE system of grading Motor Oil viscosity first was conceived in order to eliminate the uncertainty of such older terms as "light," "heavy," "medium," etc., and place a more exact specification on viscosity or body.

For many years emphasis has been put on this principal point that the SAE number refers to viscosity only and does not cover

any other psysical specification of the oil. SAE grades range from SAE 10, which is the lightest, to SAE 70, which is the heaviest.

Each of the SAE grades permits a variation of 25 or 30 seconds between the top and bottom of the viscosity range.

For instance, SAE 50 may be anything between 80 and 105 seconds Saybolt at 210°F. However, any one oil company will probably hold its SAE 50 oil at some one definite place in this range so this does not mean that a particular brand of oil may vary all the way from 80 to 105.

Aircraft manufacturers do not recommend Engine Oils with as large a variation in viscosity as is permitted under the SAE viscosity ranges. Therefore, aircraft manufacturers do not, as a rule, recommend Engine Oils by SAE numbers but by aviation grade numbers.

These aviation grade numbers are the viscosity of the oil at 210° plus or minus a slight variation usually 2½ seconds over or under the given viscosity. The aviation grades in use at the present time are 55, 60, 80, 100 and 120. This means that a grade 100 Engine Oil would normally have a viscosity of 100 seconds Saybolt at 210°F. but might go as low as 97½ seconds or as high as 102½ seconds. This would be within the viscosity range of SAE 50, but since SAE 50 permits a variation of anywhere from 80 to 105 seconds Saybolt—this is not a close enough specification for the aircraft industry.

Aircraft engine recommendations are also somewhat modified by the fact that they have not only the viscosity specifications as outlined above but are refined to a much higher degree than would be required for an automobile. Such aviation oils would have specifications set up by the refiner to make sure that they would operate satisfactorily in an airplane.

The special conditions that an airplane Engine Oil has to meet are extremes of temperature on account of altitude, operating conditions that promote the formation of lacquer, sludge, and other evils to a far greater degree in an airplane than they do in an automobile.

"AN" Specifications

The Armed Services of the United States have set up a specification for aviation Engine Oils known as "Army-Navy" specifications, or for short, "AN." These make use of much the same viscosity numbers as are used for civilian aviation Engine Oils but they have other specifications than viscosity. The "AN" oils use a series of numbers beginning with 1000 which is placed in front of the viscosity number at 210°F. An aviation Engine Oil 100 would be represented by an "AN" number 1100.

Aviation engine grade 60 has a slight difference in viscosity when it comes to an "AN" number as this is called 1065. Aviation Engine Oil grade 55 has no equivalent "AN" grade.

The "AN" specifications, in addition to viscosity, include viscosity index, oxidation number, flash point, pour point, carbon residue, and neutralization number.

A service station operator who is entirely familiar with automobile Motor Oils may be slightly confused by this new numbering system. If he remembers that the aviation grade which is part of the name automatically indicates that such oil is suitable in aviation engines, he should have no trouble if he refers to the recommendation table for that particular engine.

Special Refining

Automobile Motor Oils are not suitable for use in aviation engines except in an emergency because they do not have the special refining processes and detailed specifications to make them suitable for this purpose.

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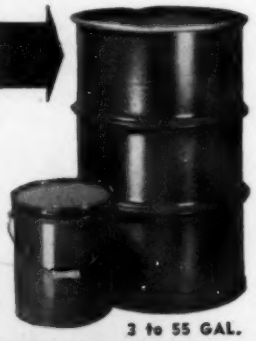

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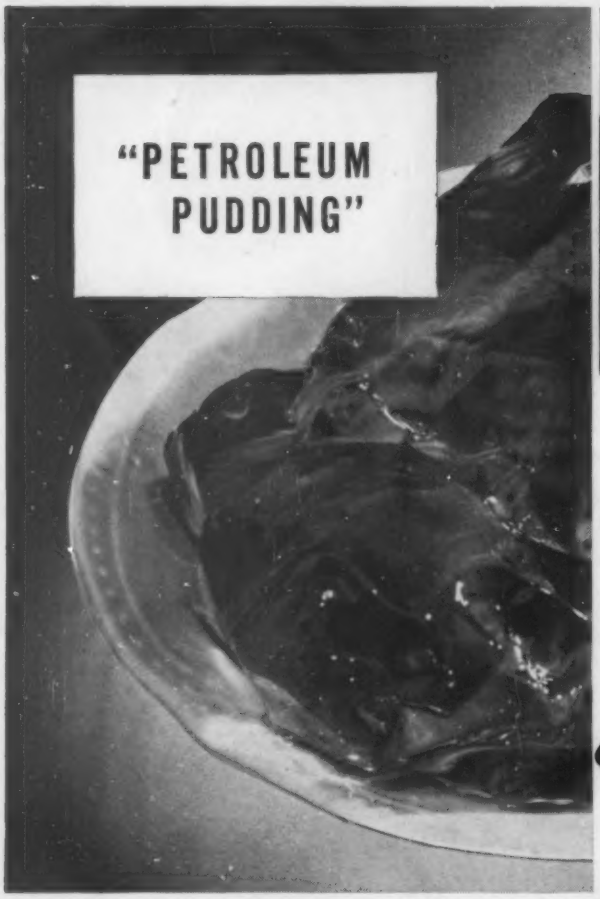

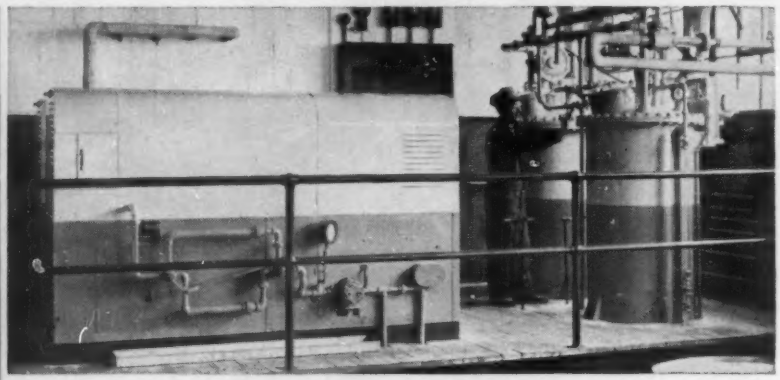

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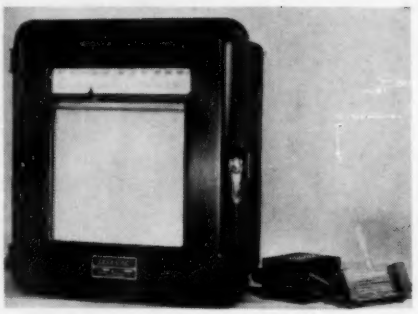

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